

This Page Is Inserted by IFW Operations  
and is not a part of the Official Record

## **BEST AVAILABLE IMAGES**

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images may include (but are not limited to):

- BLACK BORDERS
- TEXT CUT OFF AT TOP, BOTTOM OR SIDES
- FADED TEXT
- ILLEGIBLE TEXT
- SKEWED/SLANTED IMAGES
- COLORED PHOTOS
- BLACK OR VERY BLACK AND WHITE DARK PHOTOS
- GRAY SCALE DOCUMENTS

**IMAGES ARE BEST AVAILABLE COPY.**

**As rescanning documents *will not* correct images,  
please do not report the images to the  
Image Problem Mailbox.**

**THIS PAGE BLANK (USPTO)**

---

①②

**EUROPEAN PATENT APPLICATION**

②① Application number: **82300529.3**

⑤① Int. Cl.<sup>3</sup>: **C 11 D 11/02, C 11 D 17/06,**  
**C 11 D 9/10**

②② Date of filing: **02.02.82**

③③ Priority: **04.02.81 GB 8103444**

④③ Date of publication of application: **11.08.82**  
**Bulletin 82/32**

⑧④ Designated Contracting States: **AT BE CH DE FR GB IT**  
**LI NL SE**

⑦① Applicant: **UNILEVER PLC, Unilever House Blackfriars P**  
**O Box 68, London EC4P 4BQ (GB)**

⑧④ Designated Contracting States: **GB**

⑦① Applicant: **UNILEVER NV, Burgemeester s'Jacobplein 1,**  
**NL-3000 DK Rotterdam (NL)**

⑧④ Designated Contracting States: **BE CH DE FR IT LI NL**  
**SE AT**

⑦② Inventor: **Evans, Elfed Huw, Agorlys Fford Berthen,**  
**Llcswm Holywell Clwyd, Wales (GB)**  
Inventor: **Rees, John Cyril, 18 Chudleigh Close Halewood**  
**Village, Liverpool Merseyside (GB)**

⑦④ Representative: **Mole, Peter Geoffrey et al, Unilever**  
**PLC, Patent Division PO Box 31 Salisbury Square House**  
**Salisbury Square, London EC4P 4AN (GB)**

⑤④ **Soap powders and a process for their manufacture.**

⑤⑦ Spray-dried or spray-cooled soap particles are coated with water-soluble inorganic substances, at least 80% of which are of a particle size below 100 microns. This improves wetting, dispersion and dissolution characteristics. The coating is particularly appropriate to soap particles containing substantial proportions of longer chain unsaturated soaps such as those derived from oleic acid, which tend to be softer than conventional tallow/coconut-derived materials.

**EP 0 057 611 A2**

SOAP POWDERS AND A PROCESS FOR THEIR MANUFACTURE

This invention relates to soap powders and to a process for preparing them.

The wetting, dispersion and solubility characteristics of soap powders are a continuing problem to the industry and much effort has been expended over the years in improving them. For example, it is disclosed in British Patent No 907,494 that granular soap compositions need to be finely-divided to ensure rapid solution but that fines lead to clotting. This can be avoided, it is stated, by the addition of a sodium acetate or sodium benzoate anti-lumping agent in granular form. A different solution to this same problem of clotting is proposed in United States Patent No 2,940,935 in which an alkali metal xylene sulphonate and an alkali metal silicate or carbonate are incorporated into the soap-containing slurry prior to spray-drying.

We have now discovered that clotting of spray-dried soap powders can be reduced by coating the powder with a finely-divided water-soluble inorganic salt.

71E22V

Accordingly, the present invention provides spray-dried particles comprising a major proportion of water-soluble soap and having an average diameter of 300-700 microns coated with particles of an inorganic substance, characterised in that the inorganic substance is water-soluble and that at least 80% by weight of its particles have an average diameter of less than 100 microns.

Treatment of spray-dried soap particles with finely-divided inorganic salts has been suggested before, in United States Patent No 2,715,110. However the inventor found that the treatment led to no improvement in the agglomeration characteristics of the soap powder and he consequently rejected it in favour of a treatment with gaseous carbon dioxide. It is not clear why the treatment with inorganic salt was found ineffective, but we believe it to have been a consequence of the relative particle sizes of the spray-dried soap and the inorganic salt. The sizes which we specify are critical to obtaining the effect.

United States Patent No 2,592,535 also discloses the coating of soap with hygroscopic salts, such as phosphates, to reduce loss of moisture from spray-dried soap particles and consequently to reduce dusting. However the critical size relationships which we specify are not referred to in this specification either.

British Patent Nos 1,401,726 and 1,463,978 relate to particles containing major proportions of soaps and lime-soap dispersants such as ether sulphates. It was apparently found that the lime-soap dispersants adversely affected the solubility properties of the soap particles and it was proposed to counteract that by impregnating them with smectite clay particles having a size below 50 microns. However such particles are not water-soluble and so will remain in suspension in the wash liquor, which may not be desirable.

As stated above the relationship of the particle size of the water-soluble soap particle to that of the coating particle of inorganic salt is an important feature of this invention. The average diameter of the soap particles can vary from 300-700 microns, preferably 500-600 microns, in which case the average diameter of the inorganic particles is such that at least 80% by weight of them are less than 100 microns, preferably less than 50 microns. The ratio of the diameter of the soap particles to that of the coating particles is preferably from 6:1 to 8:1, most preferably 7:1.

The spray-dried soap-containing particles need not consist entirely of soap, but may contain up to 49% by weight of other soap powder components. Examples of these are detergency builders, foam boosters or foam suppressants, powder structurants, anti-oxidants, softening agents and water. Preferably the particles comprise from 60-80% by weight of soap.

The term 'spray-dried' is used herein as a convenient shorthand. Soap powders are either produced by spray-drying in which a relatively cool slurry is sprayed into a hot drying gas, or by spray-cooling, in which hotter slurry is sprayed into relatively cold drying gas, and there are a number of intermediate processes which combine both spray-drying and spray-cooling. All of these processes are included within the term 'spray-dried' as used herein.

Similarly, the term 'coated' is used herein to denote a situation where a plurality of particles of the water-soluble inorganic substance are adherent to the spray-dried soap particles. It does not necessarily imply that so many are adherent that the soap particle is completely coated. It is within the scope of the invention for it to be only partly so.

The nature of the water-soluble soap is not critical to the invention. For economic reasons it will normally be

a sodium or potassium soap, but any other cation will be satisfactory provided that it is non-toxic and does not cause unwanted side effects in the composition. The fatty acid component of the soap may be derived from mixtures of saturated and partially unsaturated fatty acids in the  $C_8$ - $C_{26}$  chain length region. Coconut oil and tallow, which are the traditional soap-making materials are preferred sources of the mixed fatty acids, the former containing predominantly  $C_{12}$  and  $C_{14}$  saturated fatty acids and the latter saturated  $C_{14}$  and  $C_{18}$  acids and mono-unsaturated  $C_{16}$  acids. However, the invention is also particularly applicable to soaps formed from fatty acid mixtures containing high proportions of unsaturated acids such as oleic acid and linoleic acid. These soaps tend to be soft and creepy when in spray-dried form and the coating of the invention is especially suitable for improving their powder properties. Sunflower seed oil is an example of an oil which contains fatty acids of this type. When the spray-dried particles contain an unsaturated fatty acid having 16 or more carbon atoms then it is preferred that the content of this acid should be 25% or more by weight of the particle.

The soap particles may also contain anionic surfactants in amounts up to 15% by weight of the particle. The nature of the anionic surfactant is not critical, any of the surfactants conventionally used or proposed for use in fabric washing powders being suitable. Examples of these are sodium alkyl benzene sulphonates, preferably  $C_{10}$ - $C_{14}$  alkyl, sodium primary and secondary alkyl sulphate, preferably  $C_{10}$ - $C_{22}$  alkyl, olefine sulphonate salts and sodium alkane sulphonates.

The soap particles may also contain nonionic surfactants in amounts up to about 30% by weight of the particles. Ethoxylated and propoxylated primary and secondary alcohols are the preferred nonionic surfactants, primary alcohols containing 7 to 25 carbon atoms

ethoxylated with from 5 to 30 moles of ethylene oxide per mole of alcohol being most preferred.

As has been said, the invention is particularly applicable to spray-dried particles which are soft, and  
5 examples of components which may make the particles soft are polyethylene glycols, ethoxylated alkanolamides, alkyl phosphoric acid esters and quaternary ammonium salts.

The water-soluble inorganic salt for coating the spray-dried soap particles is preferably present in an  
10 amount of from 3 to 25% by weight of the total soap powder formulation. The lower amount is the general level which will normally be used when the salt is only performing a coating function, whereas the upper amount is the maximum amount which will normally be used when the salt performs a  
15 function in addition to coating, such as detergency building or bleaching.

Examples of suitable salts, within which term are included oxides as well as oxyacid salts, are the water-soluble phosphates, carbonates, percarbonates,  
20 borates, perborates, sulphates and metasilicates and aluminosilicates, particularly the sodium and potassium salts. In addition to the inorganic compounds referred to above, but not as an alternative to them, certain organic compounds can be used for coating. Examples of such  
25 compounds are the salts of nitrilotriacetic acid, preferably sodium nitrilotriacetate, and tetraacetyl ethylene diamine.

---

The invention provides, in a second aspect, a process for the manufacture of spray-dried particles comprising a  
30 major proportion of a water-soluble soap inorganic substance, at least 80% of which have an average diameter of less than 100 microns, characterised by forming a mixture of the two types of particle, imparting relative motion to them and spraying them with an aqueous solution  
35 of a binder.

The nature of the binder, which ensures adhesion



between the spray-dried soap particles and the inorganic salt particles is not critical, conventional binders such as water, sodium silicate solution and sodium carboxymethyl cellulose solution being satisfactory. In the case of sodium silicate solution, 3-10% by weight based on the total soap powder composition, in the form of a 20-60% w/v solution, would be a typical amount of binder to be used.

It is possible to perform the process of the invention in a number of the conventional granulating apparatus. One such apparatus is the Eirich pan (registered trade mark) which is an inclined pan rotatable about its major axis. Another is the Schugi mixer (registered trade mark) which consists of a cylindrical chamber provided with inclined ribbon-like mixing blades rotatable about the major axis of the cylinder. Whichever of these apparatus is used, the coated particles will require drying in a fluidised bed through which warm air is pumped, so since it is necessary to have a fluidised bed apparatus available and since it is possible to carry out the coating process ab initio in a fluidised bed, it is preferred to carry out the process in that way.

In the following Example, a process of coating in accordance with the invention is described.

#### Example

Spray-dried particles containing a major proportion of soap and having the composition shown below were prepared by conventional slurry-making and spray-drying techniques in two size ranges, 450 and 350 microns. The resultant particles are subsequently referred to as 'core particles'.

	<u>Parts by Weight</u>
Sodium soap (28.5% lauric acid)	42.0
Coconut oil monoethanolamide	2.6
Magnesium sulphate	0.5
5 Ethylene diamine tetra acetic acid	0.13
Free fatty acids	2.17
Water	6.5

A mixture of finely-divided inorganic salts suitable for forming a coating on the above spray-dried particles was then prepared. The composition is shown below

	<u>Parts</u>
Sodium tripolyphosphate	12.0
Sodium carbonate	2.6
Sodium carboxymethyl cellulose	0.5
15 The core particles and the mixture of inorganic salts, together with 25 parts of sodium perborate were then placed in an Anhydro (registered trade mark) fluidised bed and sprayed with a 30% w/v aqueous solution of sodium silicate ( $\text{Na}_2\text{O}:\text{SiO}_2$ , 2:1).	

20 In a second experiment the core particles, the mixture of inorganic salts and sodium perborate were fed to a Schugi cylinder mixer and sprayed with the sodium silicate solution.

25 The coated particles obtained had good flow properties, a narrow particle size distribution and a bulk density of around 0.4 g/cc.

The wetting, dispersion and dissolution properties of the coated particles were then assessed as follows.

30 A standard weight of powder was poured onto the surface of hot water (40°C) in a hand bowl and swirled round. The resultant wash liquor was then scored for wetting (ie whether the powder was floating on the surface or had sunk), dispersion (ie whether clots had formed) and dissolution on a score of 0 to 5, 5 being the best score.

35 This scoring procedure was carried out by a panel of skilled assessors, and the scores given were averaged.

0057611

The results are shown in Table 1.

Table 1

Wetting (W), Dispersion (Dp) and  
Dissolution (Ds) Test Results

		Core Particle Size					
		450 microns			350 microns		
		W	Dp	Ds	W	Dp	Ds
10	Core Particles (uncoated)	3	3	3	3	4	4
15	Coated Particles ex cylinder mixer (Schugi)	5	5	4	5	4	4
20	ex fluidised bed (Anhydro)	5	5	4	5	4	4

It can be seen from the above results that all of the coated particles are as good as or superior to the uncoated ones in wetting, dispersing and dissolving properties. The superiority is more marked with the larger core particles, and we have evidence that this is a general trend. The increase in the wetting scores of the coated particles in accordance with the invention is especially significant because this indicates that less powder floats on the surface of the water in a clot.

CLAIMS

1. Spray-dried particles comprising a major proportion of water-soluble soap and having an average diameter of 300-700 microns coated with particles of an inorganic substance, characterised in that the inorganic substance is water-soluble and that at least 80% by weight of its particles have an average diameter of less than 100 microns.
2. Spray-dried particles according to claim 1 wherein the ratio of the diameter of the spray-dried particles to that of the particles of the inorganic substance is from 6:1 to 8:1.
3. Spray-dried particles according to claim 1 or claim 2 wherein the water-soluble soap comprises an unsaturated fatty acid having 16 or more carbon atoms.
4. Spray-dried particles according to any one of the preceding claims, wherein the organic substance is a water-soluble phosphate, carbonate, borate, sulphate or metasilicate salt.
5. Spray-dried particles according to any one of the preceding claims, wherein the particles of the inorganic substance are attached to the soap particle with the aid of a binder.
6. Spray-dried particles according to claim 5 wherein the binder comprises an aqueous solution of a sodium carboxymethyl cellulose or of sodium silicate.

**BAD ORIGINAL**

7. A process for the manufacture of spray-dried particles comprising a major proportion of a water-soluble soap inorganic substance, at least 80% of which have an average diameter of less than 100 microns, characterised by forming a mixture of the two types of particle, imparting relative motion to them and spraying them with an aqueous solution of a binder.

8. A process according to claim 7 wherein relative motion is imparted to the particles by formulation of a fluidised bed.

**THIS PAGE BLANK (USPTO)**

---